# DOWEX™ Ion Exchange Resins

Properties, Impurities and Concentrations of Regenerant Chemicals

## General

Sufficient precautions should be taken when handling, transporting or disposing of acidic or basic regenerants. Even after dilution to their operational concentrations or in the waste after regeneration, sufficient acid or base can be present to cause severe damage to mankind. Adequate protection for all parts of the body should therefore be provided whenever using these chemicals, and the manufacturer's guidelines for handling these products should be carefully followed.

The purity specifications of regenerant chemicals are designed to assure trouble-free operation of the ion exchange resin after regeneration. The chemicals, therefore, have to be free of suspended materials, or other materials that may be precipitated on, or absorbed by the resin. They should also be free of ionic species other than the active regeneration agents, as this will decrease the regeneration efficiency and/or increase the leakage of this species during the operational cycle. For example, sodium hydroxide containing 2 percent NaCl will reduce the efficiency by 5 to 10 percent and cause a higher $Cl^-$ leakage from the strongly basic anion exchange resin.

In counter-current operations where low leakage levels are especially aimed for, regenerants should contain minimal levels of impurities.

Different processes and technologies and different requirements as to the quality of the treated effluent will therefore impose different restrictions on the impurity levels in the regeneration chemicals and the dilution water. In the same way, regenerant concentrations and flow rates can affect the efficiency of the operation.

Recommendations on the quality of regeneration chemicals are given in the following sections. The recommended qualities should prove sufficient for all ion exchange resin applications, and under certain conditions lesser qualities can be used, including eventually waste chemicals from process streams. Figures for impurity levels are on the basis of a 100 percent regeneration chemical.

## Hydrochloric Acid: HCl (muriatic acid)

Both as a gas and in solution, HCl is very corrosive and can cause severe burns on contact. Mucous membranes of the eyes and of the upper respiratory tract are especially susceptible to high atmospheric concentrations. Avoid inhalation of the fumes and provide adequate ventilation when handling the acid. The acid is commercially offered as a colorless to light yellow/green liquid in concentrations of about 28 to 36 weight to weight percent HCl.

| | Recommended max. impurity levels |
|---|---|
| Fe | 0.01% |
| Other metals, total | 10 mg/l |
| Organic matter | 0.01% |
| Sulfuric acid, as $SO_3$ | 0.4% |
| Oxidants ($HNO_3$, $Cl_2$) | 5 mg/l |
| Suspended matter as turbidity | ~ 0 |
| Inhibitors | None |

Hydrochloric acid from hydrolysis of chlorinated organic materials is not suitable for use as regenerant. Acid from the salt-acid process or the hydrogen-chlorine process is satisfactory.

Hydrochloric acid solutions are most diluted to 4 to 5 percent for the regeneration of strongly acidic ion exchangers, and from 1 to 5 percent for weakly acidic resins in water demineralization applications. Higher concentrations using 8 to 10 percent HCI are sometimes preferred in other applications.

## Sulfuric Acid: $H_2SO_4$

Sulfuric acid is dangerous when improperly handled. Concentrated solutions are rapidly destructive to tissues they contact, producing severe burns. Contact with eyes will cause severe damage and blindness. Inhaling vapors from hot acid or oleum may be harmful. Swallowing may cause severe injury or death. One should be well aware of the strong exothermicity of the dilution of $H_2SO_4$ with water, which can raise the temperature very high and very fast. The acid is supplied as a colorless to yellow/brown liquid in concentrations of about 93 weight percent.

Sulfuric acid solutions are mostly diluted to 1 to 6 percent for the regeneration of strongly acidic ion exchangers and to 0.5 to 1 percent for weakly acidic ion exchangers in water demineralization applications. Stepwise increase of the acid concentration may be preferred under circumstances of high-hardness waters.

| | Recommended max. impurity levels |
|---|---|
| Fe | 50 mg/l |
| Other heavy metals | 20 mg/l |
| Organic matter | 0.01% |
| Nitrogen compounds | 20 mg/l |
| As | 0.2 mg/l |
| Suspended matter as turbidity | ~ 0 |
| Inhibitors | None |

## Sodium Hydroxide: NaOH (caustic soda)

Sodium hydroxide or caustic soda can cause severe burns on contact with skin, eyes or when taken internally. Great care must be taken when handling the anhydrous material or when preparing or handling caustic soda solutions.

Caustic soda is offered as solid flakes or pellets of about 98% NaOH or as a 30 to 50% liquid. It is mostly diluted to between 2 and 5% for the regeneration of weakly or strongly basic resins.

Typical analyses for different caustic qualities are given in the following table:

| Compound | Mercury1 grade | Rayon1 grade | Regular diaphragm grade | Regular technical flake |
|---|---|---|---|---|
| NaOH | 51% | 50.1% | 50.4% | 98% |
| $Na_2CO_3$ | 0.02% | 0.2% | 0.2% | 0.5 - 1% |
| $NaClO_3$ | 1 mg/l | 2 mg/l | 5,000 mg/l | 2 mg/l |
| NaCl | 0.002% | 0.2 - 0.5% | 1 - 2% | 0.4 - 1.5% |
| $Na_2SO_4$ | 10 mg/l | 0.1% | 0.03% | 0.3% |
| Fe | 1 mg/l | 10 mg/l | 15 mg/l | 10 mg/l |
| Heavy metals | 2 mg/l | 4 mg/l | N.S. | 2 mg/l |
| $SiO_2$ | 10 mg/l | 40 mg/l | N.S. | 500 mg/l |

Regeneration of strongly basic anion exchangers is influenced by the quality of caustic available. Chloride, chlorate and ferrate ions are potential contaminants in caustic that may affect resin regeneration efficiency and stability. Sodium chlorate itself has very little oxidative properties in neutral or alkaline solutions, but in acid conditions (i.e. during resin exhaustion), chloric acid is generated which is a powerful oxidizing agent.

Chlorates have a strong affinity for the resin and will tend to be exchanged on the resin. It is suspected that the chlorate will be eluted from the column with other anions during exhaustion and when low pH solution contacts the bed, oxidation will result.

The recommended caustic quality for ion exchangers is as follows:

| | Recommended max. impurity levels |
|---|---|
| NaOH | 49 - 51% |
| NaCl | 1.0% |
| $NaClO_3$ | 1,000 mg/l |
| $Na_2CO_3$ | 0.2% |
| Fe | 5 mg/l |
| Heavy metals (total) | 5 mg/l |
| $SiO_2$ | 50 mg/l |
| $Na_2SO_4$ | 250 mg/l |

Mercury cell or purified diaphragm cell (rayon) quality sodium hydroxide will normally meet such specifications. Regular diaphragm cell quality caustic soda can contain over 2% NaCl and over 0.1% (1000 mg/l) $NaClO_3$.

For high water quality applications with counter-current regeneration systems (such as UPCORE™ packed bed systems), mixed beds, condensate polishers and where low chlorides are required, mercury or rayon grades should be used.

Weakly basic resins will suffer mostly from high $NaClO_3$ levels as conversion to $HClO_3$ can create a strong oxidizing agent. Therefore, weak base anion exchangers should not be regenerated with regular grade diaphragm cell caustic soda. As long as chlorate and ferrate levels are low, regeneration of weakly basic resins will not suffer from high NaCl, $Na_2SO_4$ or $Na_2CO_3$ levels.

Regeneration of strongly basic resins can eventually be carried out with NaOH containing higher NaCl concentrations at the expense however, of efficiency (1% NaCl will cause about 10% reduction in efficiency). This reduction will not be noticed if a demineralizer train that is simultaneously regenerated is cation resin limited (i.e., breaks on sodium) or throughput is based on time and not leakage. Even if the anion resin breaks first, this reduction may go unnoticed since there can be other factors in a production environment that limit throughput. $NaClO_3$ levels of 1,000 mg/l can be allowed for strongly basic resins in single beds.

Studies[1] comparing the performance of DOWEX™ strong base anion resins regenerated with mercury cell caustic and regular diaphragm grade caustic in a co-flow system showed no differences in the rinse requirements or water quality produced by either caustic type. There was however a 3-6% reduction in the operating capacity of strong base resins regenerated with regular grade caustic. Subsequent field trials showed no measurable difference in resin performance and supported the fact that regular-grade caustic can be used in most co-current demineralizer systems. Many co-flow units operate today with regular grade caustic.

## Ammonia: $NH_3$

Ammonia gas or fumes from concentrated solutions can cause serious irritation to eyes and the respiratory tract. Avoid inhalation and provide adequate ventilation when handling ammonia solutions.

Ammonia is mostly offered as a solution in water, containing 20 to 30 weight percent $NH_3$. Impurities are normally minimal and cause no potential problem in ion exchange regeneration.

Ammonia is mostly used in concentrations between 3 and 5 percent for regeneration of weakly to medium basic anion exchange resins.

## Sodium Carbonate: $Na_2CO_3$ (soda ash)

Sodium carbonate does not require special handling precautions. It is supplied as a white, anhydrous powder with over 98 percent purity. Impurity levels are thus minimal and cause no potential problem in ion exchange regeneration. Moreover, higher levels of NaCl or $Na_2SO_4$ will not adversely affect the regeneration efficiency, although they will of course not contribute as regeneration chemicals.

Sodium carbonate is mostly diluted to between 5 and 8 percent for the regeneration of weakly to medium basic ion exchange resins.

## Sodium Chloride: NaCl (salt)

Sodium Chloride does not require special handling precautions. It is offered as a white powdered, granulated or pelleted solid of 98 to 99 percent.

| | Recommended max. impurity levels |
|---|---|
| $SO_4^{--}$ | 1% |
| $Mg^{++}Ca^{++}$ | 0.5% |

Sodium Chloride is used for regeneration in different processes. Concentrations will differ depending upon the process, as is illustrated in the following table.

| Process | Resin | Concentration |
|---|---|---|
| Softening | e.g., DOWEX™ MARATHON™ C | 8 - 26% NaCl |
| Dealkalization | e.g., DOWEX MARATHON A2 | 5 - 10% NaCl |
| Organic screen | e.g., DOWEX MARATHON 11 | 10% NaCl + 1% NaOH |

## References

Information on the use of regular grade NaOH to regenerate anion exchange resins can be found in the following documents:


1. *Regeneration of Anion Exchange Resins with Regular-Grade Diaphragm-Cell Caustic Soda: A Five-Year Plant Trial* (IWC Proceedings, 10/88, S.D. Coker, M.P. Murphy)
2. *Petrochemical Company Anion Exchange Resin Regeneration Trial* (Dow report, 8/89, Michael A. Smith)
3. *Effects of Chloride Ion in Diaphragm-Cell Caustic Soda on Strong Base Anion Exchange Resin Regeneration* (Ultrapure Water, 9/87, T.J. Wainerdi)
4. *Analysis of Water Quality Following Regeneration with Regular Grade Caustic Soda - Mobil Oil Corporation (Torrance, CA)* (Dow Report ICD-534-364, 12/86, Ralph Beaver, T.J. Wainerdi, R.B. Reyes)
5. *Caustic Soda for Ion Exchange Resin Regeneration* (Marketing Research report, 4/86, Ralph A. Bacon)



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